Docket No.: 27702/10054B
(PATENT)

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re Patent Application of:
Craig A. Bonda et al.

Application No.: 10/785,271 Confirmation No.: 3869

Filed: February 24, 2004 Art Unit: 1616

For: PHOTOSTABILIZATION OF A SUNSCREEN COMPOSITION WITH A COMBINATION OF AN α-CYANO-β,β-DIPHENYLACRYLATE COMPOUND AND A DIALKYL NAPHTHALATE Examiner: M. Lamm

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

## DECLARATION OF CRAIG A. BONDA

NOW comes CRAIG A. BONDA, and after being duly sworn, states as follows:

1. I am the first named inventor of the invention disclosed and claimed in the above-identified patent application.

2. I have worked in sunscreen technology since 1995, and hold 17 U.S. patents directed to sunscreen active compounds, compositions, and method of filtering UV radiation and have published or presented at least ten papers in sunscreen technology.

3. I have studied the first Office Action dated June 29, 2004, the prior art references applied against the claims of the above-identified application, and I attended the interview with Examiner Lamm on 26 August 2004 together with this assignee's patent attorney, Richard H. Anderson. At the interview of 26 August 2004, the double-patenting rejections and the prior art rejections were discussed in detail.

4. As discussed with Examiner Lamm at the interview of 26 August 2004, the Gers-Barlag et al. publication U.S. 2001/0022966 A1 ('966) discloses wide ranges

for both the octocrylene (OC) and naphthalene diester or polyester (TQ) composition components. Gers-barlag '966 discloses 4-16% by weight TQ (see paragraph [0036]), with the amount of octocrylene being advantageously less than 1% by weight (paragraph [0038]) or greater than 1% by weight (paragraph [0039]). The examples disclose the combinations of OC and TQ in a weight ratio of OC/TQ in the range of 0.16 (Ex. 6) to 0.72 (Ex. 8). There is always more TQ than octocrylene in the formulations of the '966 Gers-Barlag et al. published application, and the minimum quantity of TQ is 4.0% by weight.

5. All claims of my application call for a weight ratio of octocrylene to TQ of at least 0.95, preferably at least 1.0.

6. At the interview of 26 August 2004, the importance and non-obviousness of my claimed octocrylene/TQ weight ratio of at least 0.95 were discussed in detail. In order to show the unexpected results achieved by my claimed sunscreen formulations, in comparison to the sunscreen formulations disclosed in the Gers-Barlag et al. '966 published application, I personally conducted comparative experiments to compare my claimed formulations, having an octocrylene/TQ weight ratio of at least 0.95, to sunscreen formulations having a weight ratio of octocrylene/TQ of 0.70 and 0.72 (the highest weight ratios disclosed in the Gers-Barlag et al. '966 publication.

7. In particular, I prepared four sunscreen formulations: two Gers-Barlag formulations containing Octocrylene (OC) and diethylhexyl 2,6-naphthalate (TQ) in a ratio of 0.70 and 0.72; and two Bonda formulations containing OC and TQ in a 0.95 and 1.0 weight ratio of OC/TQ, with a total weight of octocrylene and TQ equal to 5.0% for all four formulations. Otherwise, the four formulations were identical. I prepared slides of all four formulations on Vitro-skin, and subjected spots on each slide to 35 MED at 290-400 nm, recording absorbance both before and after irradiation.

8. Summarizing the following data in Tables 1 and 2, the Gers-Balag formulation with an OC:TQ ratio of 0.72 lost 13.75% of its UVA absorbance, 7.05% of its UVB absorbance, and 9.01% of its SPF value, as shown in the data of Table 1. The Bonda (claimed) formulation with the OC:TQ ratio of 0.95 lost 5.29% of its UVA absorbance, 5.38% of its UVB absorbance, and only 2.81% of its SPF value, as shown in Table 2.

**TABLE 1**

Labsphere Ultraviolet Transmittance Analyzer Report

**Gers-Barlag** UVB Photostability

**OC:TQ Wt. Ratio 0.72 (% octocrylene; % TQ)**

UV exposure: 35 MED
Wavelength Range: 290 - 400 nm

| Units: | SPF | T(UVA) | T(UVB) |
|---|---|---|---|
| # of Scans: | 2 | 2 | 2 |
| Mean: | 21.6 | 9.41% | 5.13% |
| STD: | 1.9 | 1.29% | 0.36% |
| % loss | 9.01% | 13.75% | 7.05% |
| Scan# | SPF | Critical Wavelength | |
| 1 | 22.98 | 381 nm | |
| 2 | 20.23 | 380 nm | |

**TABLE 2**

Labsphere Ultraviolet Transmittance Analyzer Report

**BONDA** UVB Photostability

**OC:TQ Wt. Ratio 0.95 (% octocrylene; % TQ)**

UV exposure: 35 med
Wavelength Range: 290 - 400 nm

| Units: | SPF | T(UVA) | T(UVB) |
|---|---|---|---|
| # of Scans: | 2 | 2 | 2 |
| Mean: | 26.63.8 | 6.06% | 4.42% |
| STD: | 0.75 | 0.32% | 0.24% |
| % loss | 2.81% | **5.29%** | **5.38%** |
| Scan# | SPF | Critical Wavelength | |
| 1 | 27.16 | 383 nm | |
| 2 | 26.10 | 383 nm | |

9. The comparative data for the Gers-Barlag weight ratio of octocrylene/TQ of 0.70 (Example 4) is shown in Table 3 below, and the data for my preferred weight ratio of 1.0 is shown in Table 4 below. Summarizing, the Gers-Barlag formulation with an OC:TQ ratio of 0.70 lost 12.81% of its UVA absorbance, 8.14% of its UVB absorbance, and 11.8% of its SPF value. The Bonda (claimed) formulation with the OC:TQ ratio of 1.0 lost only 7.49% of its UVA absorbance, 2.07% of its UVB absorbance, and only

3.42% of its SPF value. The data in Tables 3 and 4 again shows unexpected results for my claimed octocrylene/TQ weight ratio of 1.0 compared to the octocrylene/TQ weight ratio of 0.7 of the Gers-Barlag '966 reference.

**TABLE 3**

Labsphere Ultraviolet Transmittance Analyzer Report

**Gers-Barlag**

**OC:TQ Wt. Ratio 0.7 (2.06% octocrylene; 2.94% TQ)**

UV exposure: 35 med
Wavelength Range: 290 - 400 nm

| Units: | SPF | T(UVA) | T(UVB) |
|---|---|---|---|
| # of Scans: | 2 | 2 | 2 |
| Mean: | 25.1 | 8.30% | 4.42% |
| STD: | 2.9 | 1.06% | 0.36% |
| % loss | 11.48% | 12.81% | 8.14% |

| Scan# | SPF | Critical Wavelength |
|---|---|---|
| 1 | 27.12 | 382 nm |
| 2 | 23.05 | 381 nm |

loses > 4 SPF

**TABLE 4**

Labsphere Ultraviolet transmittance Analyzer Report

**BONDA**

**OC:TQ Wt. Ratio 1 (2.5% octocrylene; 2.5% TQ)**

*(Note: This formulation was prepared 10/09/03, nearly a year before this study was conducted. The three other formulations represented in this declaration were freshly prepared within days of being tested. Fresh formulas may perform better.)*

UV exposure: 35 med
Wavelength Range: 290 - 400 nm

| Units: | SPF | T(UVA) | T(UVB) |
|---|---|---|---|
| # of Scans: | 2 | 2 | 2 |
| Mean: | 20.0 | 8.06% | 6.03% |
| STD: | 0.7 | 0.60% | 0.12% |
| % loss | 3.42% | 7.49% | 2.07% |

| Scan# | SPF | Critical Wavelength |
|---|---|---|
| 1 | 20.48 | 382 nm |
| 2 | 19.52 | 381 nm |

10. The above results were most unexpected to me and are unexpected to others skilled in the sunscreen art.

11. All statements made herein of my own knowledge are true, and that all statements made upon information and belief are believed to be true, and further that these statements are made with the knowledge that willful false statements and the like, so made are punishable by fine or imprisonment, or both, under section 1001 of title 10 of the United States Code and that such willful false statements may jeopardize the validity of the instant patent application or any patent issuing thereon.

Dated: September 22, 2004

Craig A. Bonda

# Consumer Product Testing Co.

EST. 1975

## FINAL REPORT

**CLIENT:** The C.P. Hall Company
5851 West 73rd Street
Chicago, Illinois 60638

**ATTENTION:** Craig Bonda
Director, R&D – Personal Care

**TEST:** Sunscreen PFA Testing
Protocol: 7.06

**TEST MATERIAL:** CAB4-269
OC/DEHN = 1

**EXPERIMENT REFERENCE NUMBER:** S03-1221

Maryann Ackerman
Maryann Ackerman
Quality Assurance Associate

R. W. Shanahan
Robert W. Shanahan, Ph.D.
Vice President, Technology

Caryl K. Wood
Caryl K. Wood
Director of Photobiology





70 New Dutch Lane • Fairfield, New Jersey 07004-2514 • (973) 808-7111 • Fax (

Consumer Product Testing Co.
EST. 1975

# QUALITY ASSURANCE UNIT STATEMENT

**Study No.:** S03-1221

The objective of the Quality Assurance Unit (QAU) is to monitor the conduct and reporting of clinical laboratory studies. These studies have been performed with adherence to ICH Guideline E6 for Good Clinical Practice and requirements provided for in 21 CFR parts 50 and 56 and in accordance to standard operating procedures and applicable protocols. The QAU maintains copies of study protocols and standard operating procedures and has inspected this study on the date(s) listed below. The findings of these inspections have been reported to management and the Study Director. All materials and data pertinent to this study will be stored in the Archive Facility at 70 New Dutch Lane, Fairfield, New Jersey, 07004, unless specified otherwise, in writing by the Sponsor.

**Senior personnel involved:**

| | | |
|---|---|---|
| Caryl K. Wood | - | Director of Photobiology |
| Robert W. Shanahan, Ph.D. | - | Vice President, Technology |
| Michael Lutz, B.S. | - | Manager, Technical Services |
| Maryann Ackerman | - | Quality Assurance Associate |

The representative signature of the Quality Assurance Unit signifies that this study has been performed in accordance with standard operating procedures and the applicable study protocol as well as any government regulations regarding such procedures and protocols.

**Objective:** To estimate (in-vivo) the level of protection afforded by a sunscreen product against UVA radiation using Persistent Pigment Darkening (PPD) as a visual end point.

**Test Sample:** CAB4-269
(expected PFA = 8)

**Control Sample:** A control standard formulated to provide an approximate Protection Factor of UVA (PFA) of 3.75 was run concurrently with the test material.

**Study Schedule:**

| Initiation Date | Completion Date |
|---|---|
| October 31, 2003 | November 3, 2003 |

**Reference:** The test procedure was based on the Japanese Cosmetic Industry Association (JCIA) recommended method for UVA Protection based on Persistent Pigment Darkening.

**Inclusion Criteria:** Healthy male or female volunteers:

a) 18 to 60 years of age;

b) With Skin Types II - IV, determined by the following guidelines:

| Skin Type | Sunburn and Tanning History |
|---|---|
| I | Always burns easily; never tans (sensitive) |
| II | Always burns easily; tans minimally (sensitive) |
| III | Burns moderately; tans gradually (normal) |
| IV | Burns minimally; always tans well (normal) |
| V | Rarely burns; tans profusely (insensitive) |
| VI | Never burns; deeply pigmented (insensitive) |

c) Dependable and capable of following directions;

d) Having completed a Medical History form;

**Inclusion Criteria (continued):**

e) Having read, understood and signed an Informed Consent form.

**Exclusion Criteria:**

a) Subjects with a history of abnormal response to sunlight;

b) Subjects exhibiting current sunburn, suntan, or uneven skin tone which might be confused with a reaction from the test material or interfere with the evaluation of test results;

c) Pregnant or lactating females;

d) Subjects taking medication which might produce an abnormal response to sunlight or interfere with the results of the test;

e) Subjects who regularly use UVA tanning beds; or

f) Subjects exhibiting any visible skin disease which could be considered to affect the purpose or integrity of the study.

**Test Method:**

Five (5) subjects who met the inclusion criteria were selected for participation.

**Light Source:** A Xenon Arc Solar Simulator* (150w) was used as the source of ultraviolet light. A continuous emission spectrum in the UVA range (320-400 nanometers), using Schott WG 335/2 mm and UG11/1 mm filters, was produced during the testing procedure by this instrument.

**Determination of Minimal Persistent Pigment Dose (MPPD):** The MPPD is defined as the time interval or dosage of UVA light exposure sufficient to produce defined pigment darkening on designated test sites. Prior to the testing phase, the MPPD of the unprotected skin of each subject was determined by a progressive sequence of timed UVA light exposures, graduated incrementally by 25% over that of the previous exposure. Persistent Pigment Darkening (PPD) values were determined by visual examination of the sites made 120, 180, and 240 min. after irradiation using the following scoring system:

*Manufactured by Solar Light Company, Philadelphia, PA.

**Test Method (continued):**

0 = Negative, no visible pigment darkening
0.5 = Minimal pigment darkening
1.0 = Defined pigment darkening
2.0 = Moderate pigment darkening
3.0 = Marked pigment darkening

**Determination of PFA:** A sufficient number of test site areas were outlined with a surgical marking pen on the subject's back between the scapulae and the beltline, lateral to the midline. These areas were designated for the Test Material and Standard, with an adjacent site designated for a concurrent MPPD determination (unprotected control).

A 2mg/cm$^2$ portion of the Test Material and of the Standard was applied to the appropriate designated test site and spread evenly over the site using a fingercot. After product application, the test areas were subdivided into sites which were used for serial UVA light exposure. Irradiation of the sites was begun no less than 15 minutes and no longer than 30 minutes after application.

Exposure times were selected for each sub-site based upon the previously determined MPPD of the **unprotected** skin and the expected PFA value of the Test Article or the Standard.

All test sites were evaluated at 120, 180, and 240 minutes, post-irradiation to determine Minimal Persistent Pigment Darkening.

**Calculation of the PFA** - The PFA for the Test Material and Standard was calculated as follows:

$$PFA = \frac{\text{MPPD Test Material or Standard}}{\text{MPPD Unprotected Skin}}$$

**Sunscreen Category:** For labeling purposes, a sunscreen product may be categorized as follows:

| PFA Value | PA (Protection grade of UVA) |
|---|---|
| Over 2, less than 4 | PA+ |
| 4 or more, less than 8 | PA++ |
| 8 or more | PA+++ |

**Test Test Results:**

Results are based on five (5) subjects.

PFA calculations for each subject are represented in Table 1. No adverse dermal effects were observed on the treated areas of any subject.

**Conclusion:**

Under the test conditions of this study, Test Material: CAB4-269 exhibited an average PFA value of 8.63 at 180 minutes.

**Table 1**
**Individual PFA Values**

| Subject | CPTC# | Skin Type | Age/ Sex | Standard 180 Minutes |
|---|---|---|---|---|
| 1) JA | 38973 | IV | 54/M | 5.86 |
| 2) BB | 38974 | III | 54/F | 4.68 |
| 3) RA | 38912 | III | 26/M | 5.86 |
| 4) TF | 40813 | III | 19/M | 4.69 |
| 5) NF | 12775 | III | 56/M | 4.69 |
| **Average PFA (N=5)** | | | | 5.16 |
| **(95% Confidence Limits)** | | | | (4.36 – 5.96) |
| **Standard Deviation** | | | | 0.64 |
| **Standard Error** | | | | 0.29 |

| Subject | CPTC# | Skin Type | Age/ Sex | CAB4-269 180 Minutes |
|---|---|---|---|---|
| 1) JA | 38973 | IV | 54/M | 9.38 |
| 2) BB | 38974 | III | 54/F | 7.49 |
| 3) RA | 38912 | III | 26/M | 9.38 |
| 4) TF | 40813 | III | 19/M | 7.50 |
| 5) NF | 12775 | III | 56/M | 9.38 |
| **Average PFA (N=5)** | | | | 8.63 |
| **(95% Confidence Limits)** | | | | (7.35 – 9.91) |
| **Standard Deviation** | | | | 1.03 |
| **Standard Error** | | | | 0.46 |

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | **CAB4-269** | | | | | | | | | | |
| | Estee Lauder Project | | | | | | | | | | |
| | Revise CAB4-268 Test of OC-TQ blend above BDF patent ratio: SPF 15, take out Oxybenzone | | | | | | | | | | |
| | **Calculation of Required HLB** | | | | | | | | | | |
| | | | | | | | | | | | |
| | Ingredient | REQUIRED HLB | % of Total/100 | HLB Contribution | x5 | x15 | | Solvency | | | |
| | Avobenzone | 10 | 0.0300 | 1.13 | 15.00 | 45 | 11.26% | | | | |
| x | Octyl salicylate | 12 | 0.0500 | 2.25 | 25.00 | 75 | 18.76% | 0.01 | | | |
| x | Homosalate | 12 | 0.0750 | 3.38 | 37.50 | 112.5 | 28.14% | 0.015 | | | |
| x | Diethylhexyl 2,6-naphthalate | 12 | 0.0250 | 1.13 | 12.50 | 37.5 | 9.38% | 0.0045 | | | |
| x | Octocrylene | 8 | 0.0250 | 0.75 | 12.50 | 37.5 | 9.38% | 0.005 | | | |
| x | Dimethyl capramide | 7 | 0.0100 | 0.26 | 5.00 | 15 | 3.75% | 0.004 | | | |
| x | Diethylhexyl malate | 11 | 0.0201 | 0.83 | 10.05 | 30.15 | 7.54% | 0.00402 | | | |
| | Benzophenone-3 | 10 | 0.0049 | 0.18 | 2.45 | 7.35 | 1.84% | | | | |
| | Dimethicone | 10 | 0.0040 | 0.15 | 2.00 | 6 | 1.50% | | | | |
| | Silica (R972) | 10 | 0.0025 | 0.09 | 1.25 | 3.75 | 0.94% | | | | |
| x | Ganex V-220 | 10 | 0.0200 | 0.75 | 10.00 | 30 | | | | | |
| | Total Oil Ingredients | | 0.2665 | | 133.25 | 399.75 | 92.50% | 0.04252 | | | |
| | HLB Required | | | 10.90 | | | | | | | |
| | | | | | | | | | | | |
| | (Calculate TEA, stearate, stearic) | | | | | | | | | | |
| x | Stearic acid | | 0.0305 | | 15.25 | | | | | | |
| | TEA | | 0.0169 | | 8.46375 | | | | | | |
| | **Calculation of Surfactant HLB** | | | | | | | | | | |
| | Stearic acid | 1 | 0.0003 | 0.00 | 0.13616 | | | | | | |
| | TEA stearate | 20 | 0.0472 | 8.03 | 23.5776 | | | | | | |
| x | Sorbitan isostearate | 4.7 | 0.0400 | 1.60 | 20.00 | | | | | | |
| x | Polyglyceryl 3 distearate | 5 | 0.0300 | 1.28 | 15.00 | | | | | | |
| | Total surfactant | | 0.1174 | | | | | | | | |
| | Surfactant HLB | | | 10.91 | | | | | | | |
| | Surfactant as % of Oil Phase | | 30.59% | | | | | | | | |
| | | | | | | | | | | | |
| | **Water Phase (Exclusive of surfactants listed above)** | | | | | | | | | | |
| x | Carbomer Ultrez | | 0.0005 | | 0.25 | | | | | | |
| x | Glycerin | | 0.03 | | 15 | | | | | | |
| x | Methylpropanediol | | 0.02 | | 10 | | | | | | |
| x | Phenonip | | 0.006 | | 3 | | | | | | |
| x | Disodium EDTA | | 0.0005 | | 0.25 | | | | | | |
| | Total | | 0.0565 | | 28.25 | | | | | | |
| | **Other Ingredients** | | | | | | | | | | |
| x | TEA | | 0.0005 | | 0.25 | 8.71375 | TEA | | | | |
| | Total other ingredients | | 0.0005 | | | | | | | | |
| | Water | | 0.5591 | | 279.536 | | | | | | |
| | 1. Blended oil phase ingredients, including emulsifiers. Stirred and heated to 80C. | | | | | | | | | | |
| | 2. Dissolved EDTA in water. Added carbomer to water with stirring. Heated water to 85C. | | | | | | | | | | |
| | 3. Just before emulsification, added silica to oil phase. Stirred until wet. | | | | | | | | | | |
| | 3. Just before emulsification, added pre-blended glycols, preservative, and TEA to water. Added oil to water when oil at 85C and water at 83C. Prop. Stirred for 5 mi | | | | | | | | | | |
| | 4. Homogenized with fine screen for 3 minutes. | | | | | | | | | | |
| | 5 Sweep stirred while cooling to below 45C. | | | | | | | | | | |
| | 6. Packaged at 30-35C. | | | | | | | | | | |